General Guide to the Linkam DSC600 Stage

APS Detector/Equipment Pool

Table of Contents

Reference: much of the information in this guide is gleaned from User Manuals provided by Linkam with purchase of equipment. If more information is needed, please contact Detector Pool staff at 2-9493 or dp@aps.anl.gov.

The Linkam DSC600 is a differentially scanning calorimeter, often used in phase change experiments. The Controller collects the difference between the PT 100 temperature sensor in the silver block and the DSC sensor. This information can be used to calculate sample enthalpy. The stage can be used to heat or cool a sample from -196^{o}C to 600^{o}C at speeds of 1-100^{o}C per minute.

Quick Guide - 1

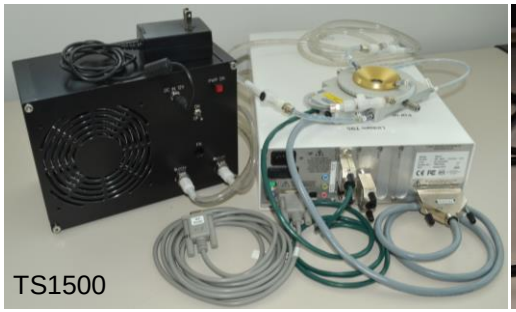
Safety & Handling

- Linkam stages have **EXTREMELY** fragile platinum leads; please do not touch or move.
- When heating above 300°C, stages require water cooling. Contact DP staff (2-9490).
- Regarding gas purging:
  - Do not use hydrogen or helium (thermal conductivity)
  - Dilute any reactive gases with 95% inert gas
  - Use only mixtures containing less than 20% oxygen
  - Flow less than 60 CC/min
- If using LN2 sample cooling, please follow all APS procedures for safe handling of LN2.

- The black capillary tube on the LN2 dewar lid is fragile; please handle carefully.
- Disconnect the LN2 dewar from the stage before heating above 300°C.

Hardware Operation

- Make all connections



- If cooling sample with LN2:
  - Fill the dewar approximately 2/3 full.
  - The LNP95 must be switched on before the T95/CI95 system controller.
  - The stage chamber needs to be purged of air before starting a cooling experiment.

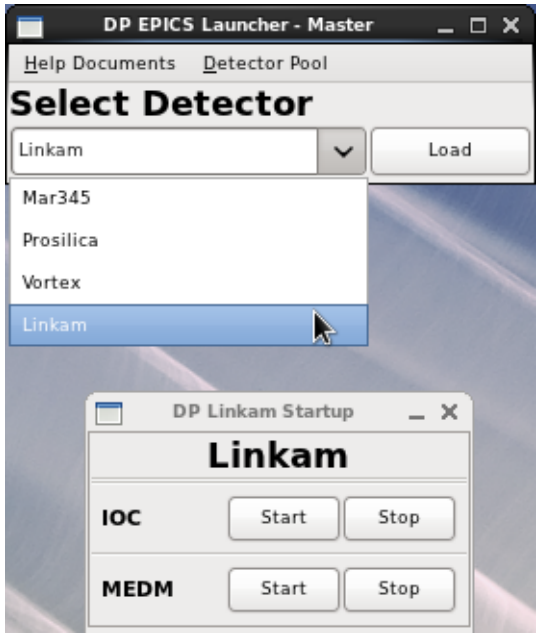
Please review the complete User Guide to ensure safe and effective operation.

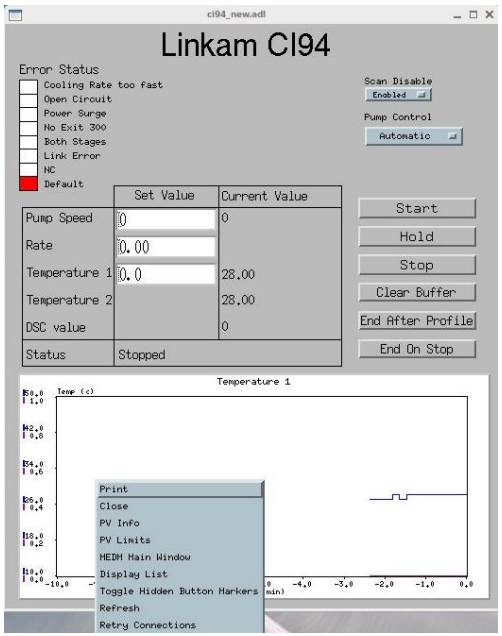
(over)

Quick Guide - 2

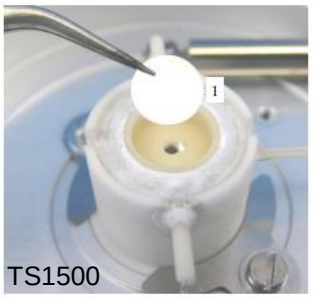
Software Operation

- Turn on the computer, and log in
  - Username = dpuser
  - Contact DP staff for the password
  - Or, use the sector's LDAP account
- The start-up screen (lower left) will appear
  - Select "Linkam from the dropdown menu
  - Click "Start" to start the IOC and medm
- Use the EPICS control screen (lower right) to set rate (°C/min) and desired temperature.
  - Pump speed is generally set to "Automatic."
  - To adjust the y-axis on the strip chart display, right-click, then select "PV Limits."

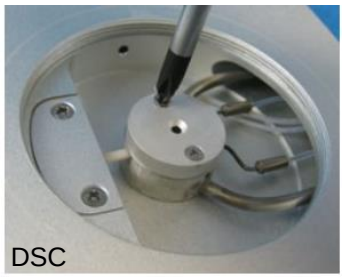


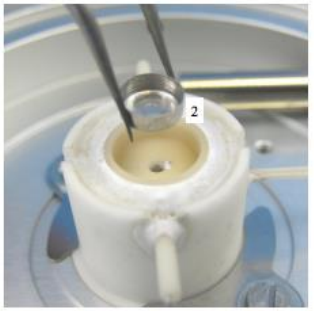


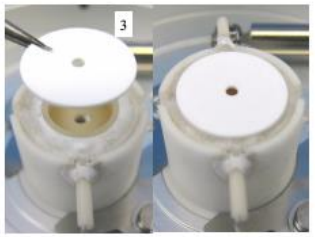
Sample Loading

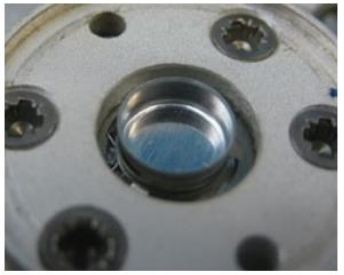


TS1500


DSC







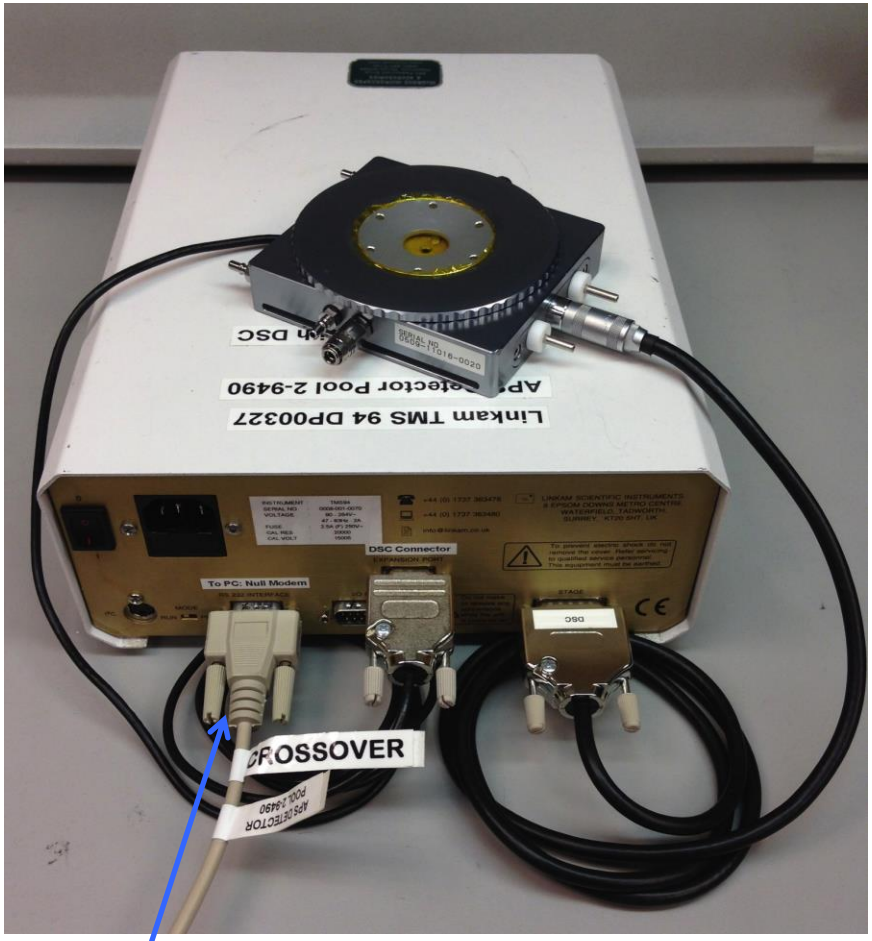

Aluminium and sapphire crucibles

Tzero Press, often used with DSC

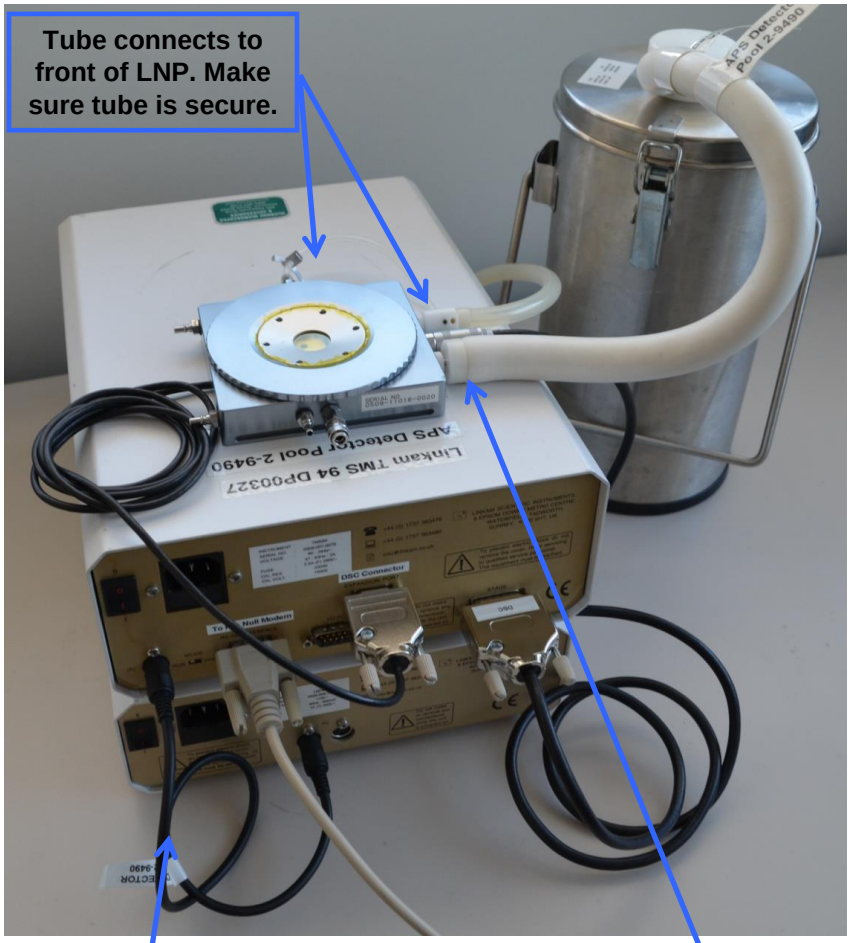
Safety and Handling

- The heating element may be fragile. Exercise care when loading and unloading samples.
- Use as little sample as possible to reduce thermal load and avoid damaging the heating element.
- Heating over 300°C
  - Stages require ***water cooling*** when heated above 300°C. Contact DP staff (2-9490 or dp@aps.anl.gov) to obtain water circulator.
  - Disconnect the LN2 dewar from the stage before heating above 300°C. Leaving the tubing connected can damage the tubing and/or heater.
- Regarding gas purging:
  - Do not use hydrogen or helium (due to high thermal conductivity)
  - Do not use a gas mixture containing more than 20% oxygen
  - Dilute any reactive gases with 95% inert gas such as nitrogen or argon
  - Use a gas flow of less than 60 CC/min to avoid overloading the heating element
- Failure to observe these guidelines may result in heating element failure.
  - If you plan to use the stage in a manner not recommended above, please purchase your own heating element.
- If using LN2 sample cooling, please follow all APS procedures for safe handling of LN2.

Connections for DSC600 with and without cooling



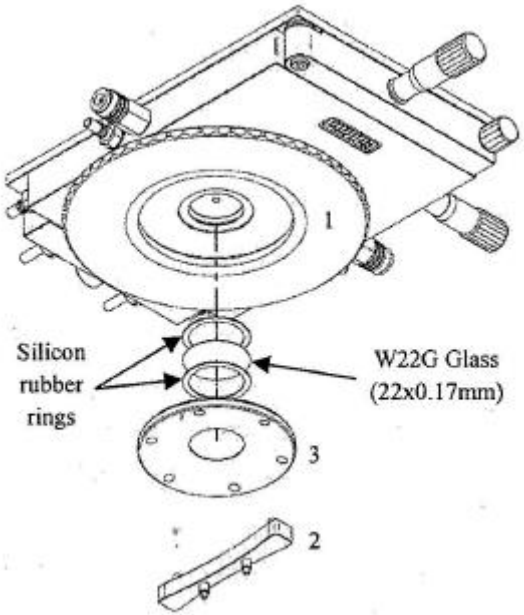
Crossover/null modem cable connects to computer serial port.



DSC600 Parts

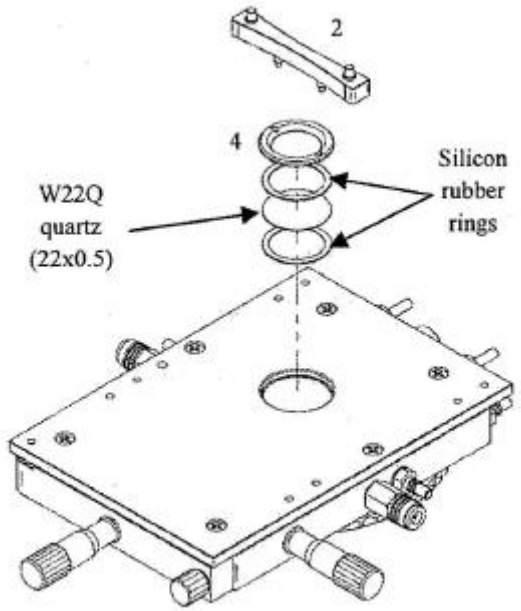
Lid Window Assembly

To replace the windows in the Stage Lid (1) use the Window Tool (2) and align the two wide spacing pins to the Tube Clip Holder holes and unscrew the Lid Insert (3).
The Stage Lid and Lid Insert should be turned upside down as shown in the diagram opposite and reassembled in the order indicated.
The Lid Insert should be screwed down until the cover slips are held firmly, then turn the assembly over and screw down the Lid Insert until it is felt to come to a stop.



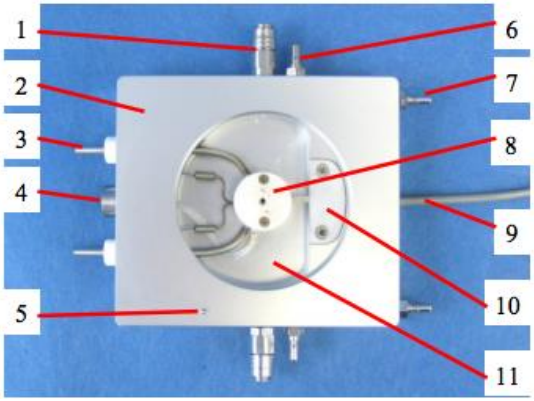
Bottom Window Assembly

Use two narrow spacing pins of Window Tool (2) to align it to the two holes of Window Locking Ring (4) and unscrew.
Reassemble the bottom window as shown in the opposite diagram

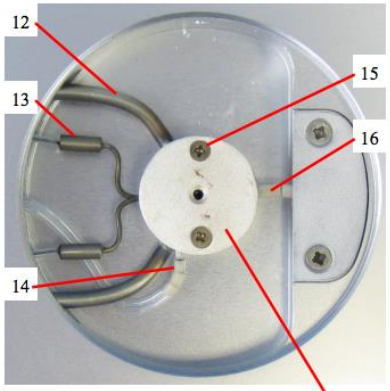


Stage Assembly

1. Gas purge valve
2. Stage Body
3. Liquid nitrogen cooling connector
4. Lemo connector for stage lead
5. Earth safety contact for lid
6. Stage body water connector
7. Bypass stage body water cooling connector
8. DSC cell carrier assembly
9. DSC cable
10. DSC sensor cable cover
11. Stage Chamber



12. Stainless steel cooling tube
13. Heating element wire
14. Platinum temperature sensor
15. Screw for DSC Cell lid
16. DSC sensor cable

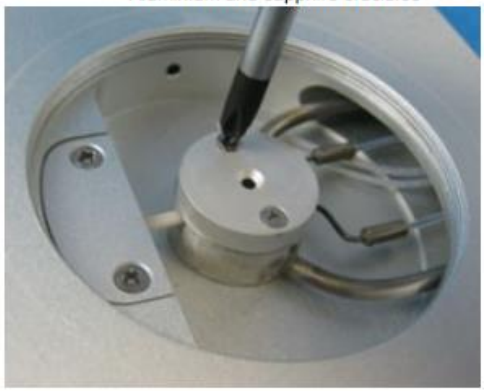


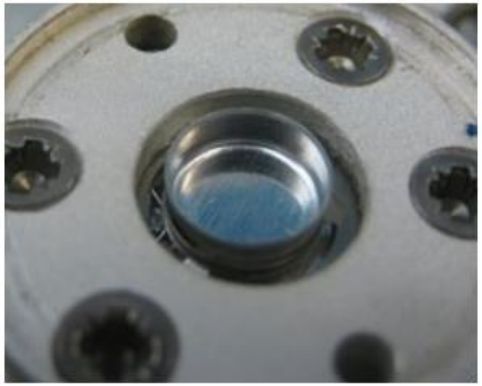
Photos/graphics courtesy of linkam.co.uk

Sample Loading

- Remove the lid of the silver chamber using a small Phillips head screw driver.
- Place the calibration crucible inside the silver chamber, making sure that the crucible does not touch the sides.
- Use very little force when screwing the lid of the cell back in position.
- For sample preparation, many users prefer the Tzero press, available through the Detector Pool.

Aluminium and sapphire crucibles





Photos/graphics courtesy of linkam.co.uk, tainstruments.com

Sample Cooling with LNP95

- If using LN2 cooling, please follow all APS procedures for safe handling of LN2.
- The black capillary tube on the dewar lid is fragile. Please handle carefully.
- Fill the dewar approximately 2/3 full.
- The LNP95 must be switched on before the T95/CI95 system controller.
- The stage chamber needs to be purged of air before starting a cooling experiment.
  - See purging procedure on next page
- Disconnect the LN2 dewar from the stage before heating above 300^{o}C. Leaving the tubing connected can damage the tube and/or heater.

Photo courtesy of linkam.co.uk

Purging Procedure

- For full details, please see the THMS600 User Guide from Linkam, available on the Detector Pool website.
- To purge using recycled nitrogen gas produced from the Dewar:
  - Make all connections, make sure that the stage lid and gas inserts are secure, turn on the LNP95, then turn on the T95/CI95.
  - Set the LNP95 to “Manual” mode.
  - Set the T95/CI95 to hold at 40ºC.
  - Set the LNP95 to a speed of 100.
  - Block the hole in the white plastic pump connector, and pinch the narrow window tube to block it.
  - Block the gas outlet for a few seconds to allow pressure to build, then release. Repeat for a few minutes.
  - When finished, unblock the pump connector, window, tube, and gas outlets.
  - Change the LNP95 to “Automatic” mode.

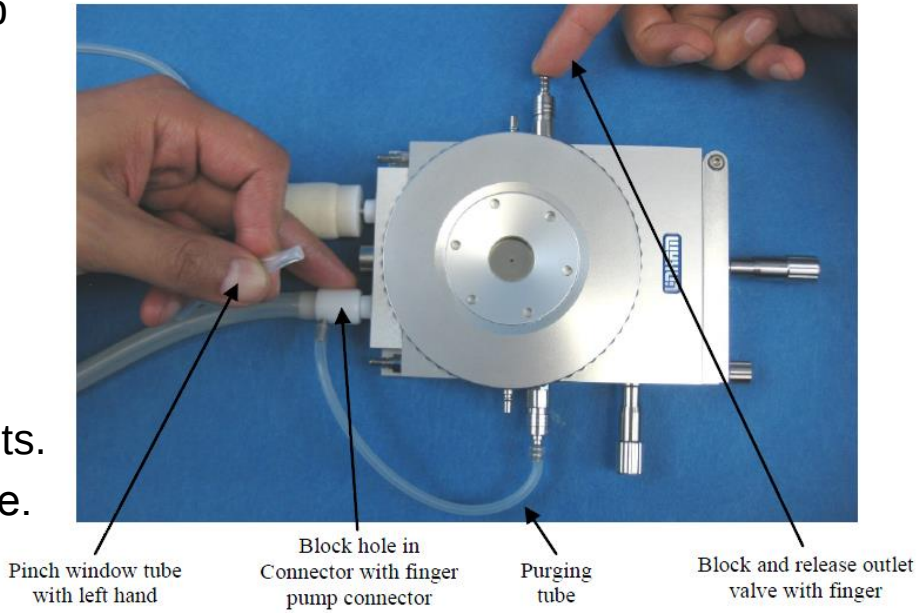
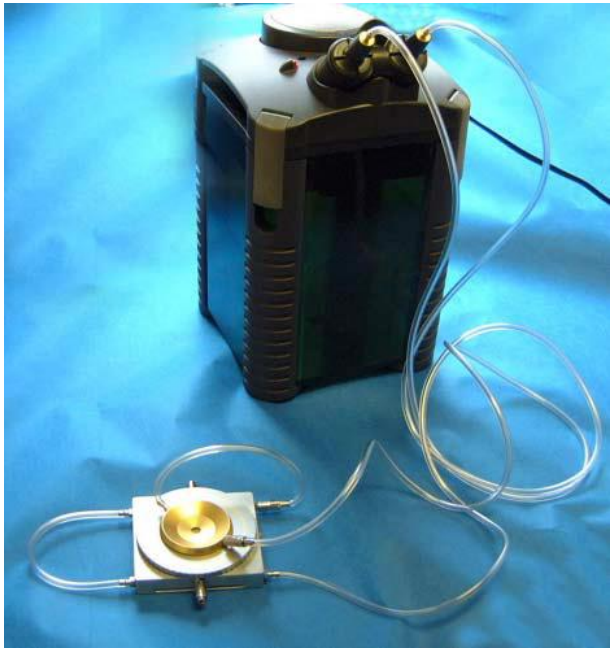


Photo courtesy of linkam.co.uk

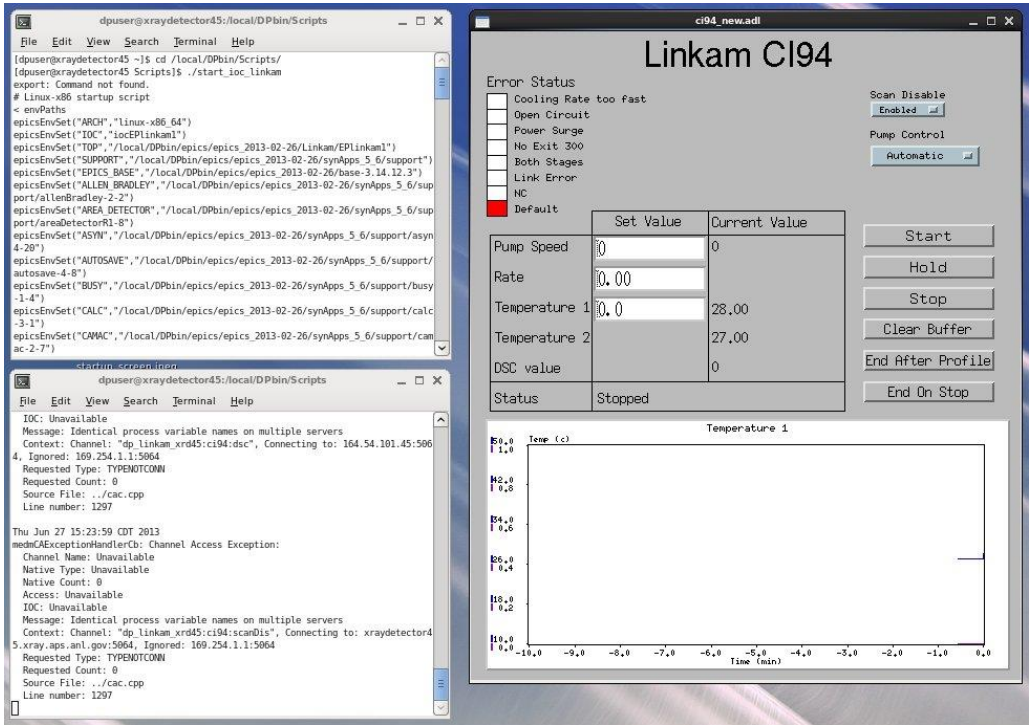
Water Cooling Connections



- When heating above 300ºC, stages require water cooling to keep the stage body and lid cool. Contact DP staff (2-9490; dp@aps.anl.gov) for a water circulator.
- There are several options for cooling:
  - Linkam ECP Water Circulating Pump
    - It may be necessary to prime the ECP if the water connectors have been removed.
  - DP circulating pump with custom housing
    - To ensure proper circulation, be careful to avoid introducing air into the system. While connecting and disconnecting, take care to prevent water from leaking out.
  - Beamlines may use their own small, recirculating chiller. (less than 20CC/min flow rate)

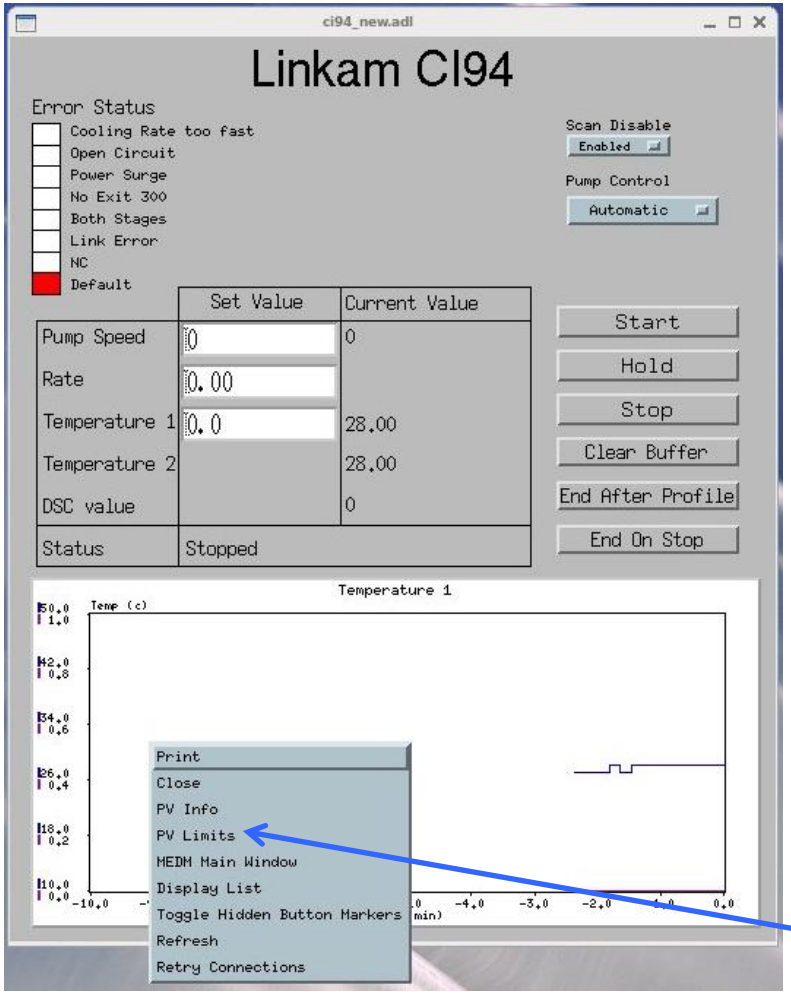
EPICS Software Controls

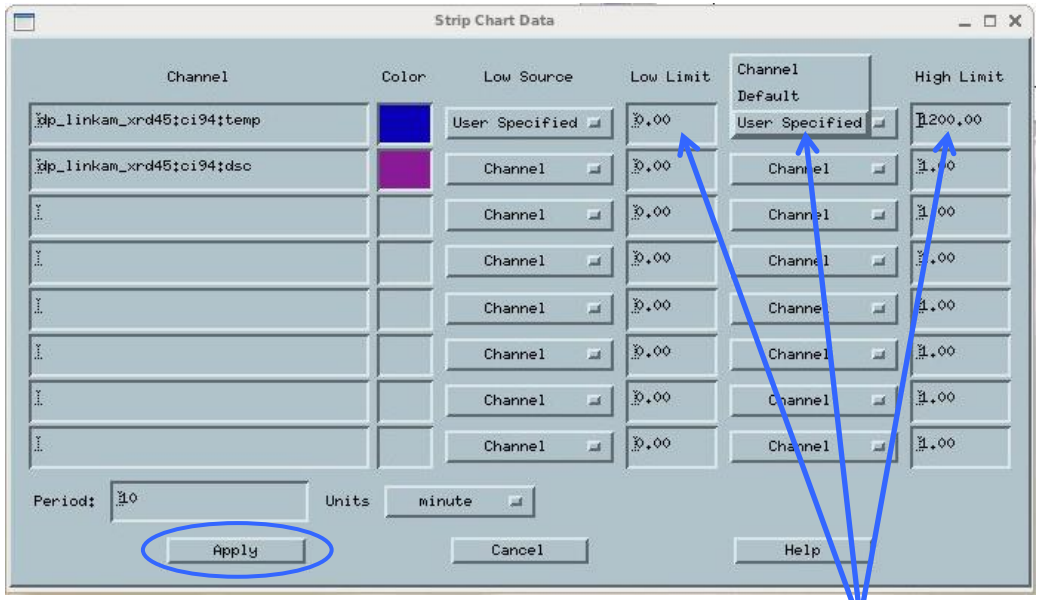
- Turn on the computer, and log in.
  - Username = dpuser
  - Contact DP staff for the password
  - Or, use the sector's LDAP account (see beamline personnel)
- The start-up screen (shown right) will appear.
  - Select "Linkam from the dropdown menu
  - Click "Start" to start the IOC and medm
- The figure below shows the two resulting terminal windows and the Linkam control screen.



- The following parameters can be set (left side):
  - Pump speed for LN2 cooling (optional—this can also be controlled automatically based on the selected cooling rate). Upper limit = 30
  - Rate of temperature change (°C/min)
  - Desired temperature
- Note the control buttons (right side):
  - Scan Disable: tells VME (EPICS sscan) to stop communicating with the controller (e.g. software is loaded but controller is not in use). "Enable" restarts communication.
  - Toggle LNP between automatic & manual
  - Start, hold, or stop heating/cooling

EPICS Software Controls





To adjust the scale on the Y-axis: right-click, then select "PV Limits."

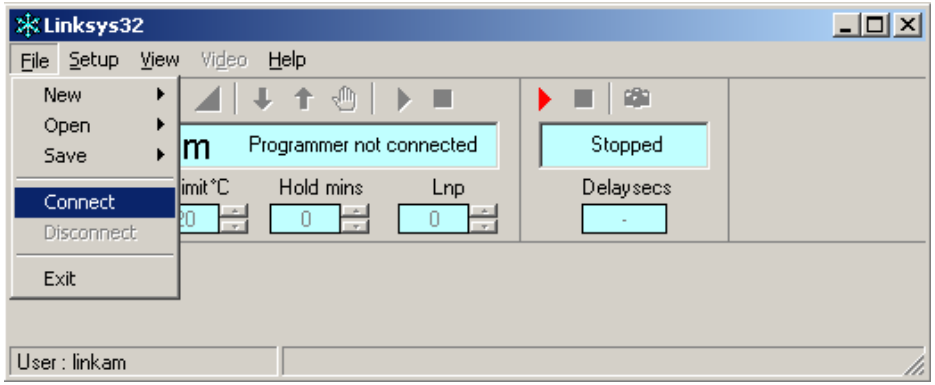
Select "User Specified," and set the high and low limits. Click "Apply."

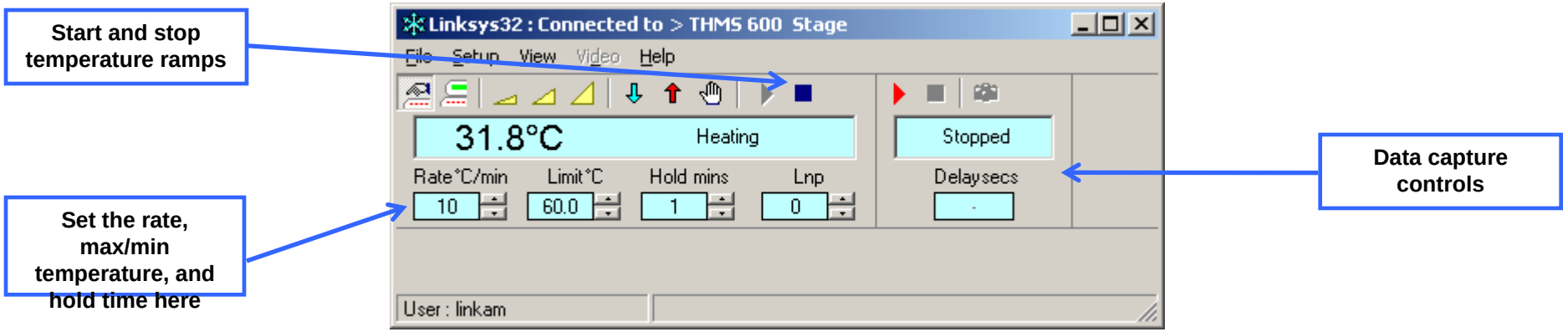
Thanks to John Hammonds for the EPICS interface!

Appendix: Linksys Software Controls

- The Linkam equipment can be controlled with either EPICS or vendor software called Linksys, but not both simultaneously.
  - The Detector Pool no longer supports the Linksys software, but some beamlines may have it installed on their computers.
  - To use the Linkam software, make sure that the EPICS IOC is not running.
- From the desktop, click Linksys32 icon

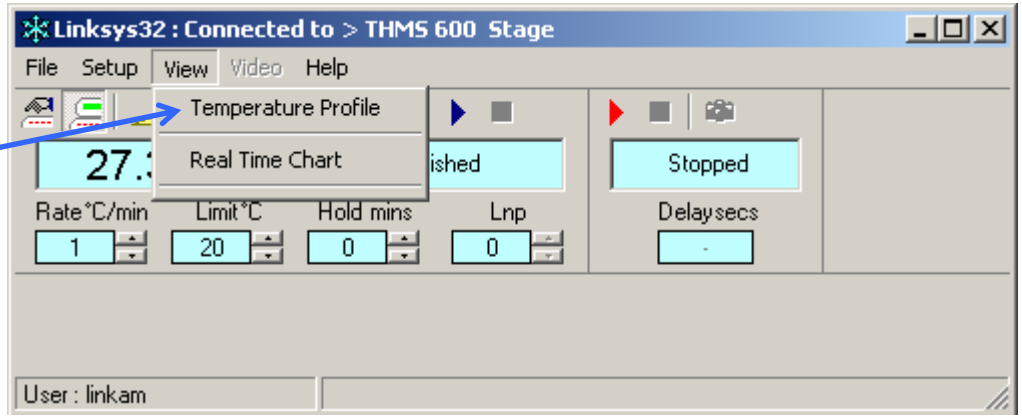
- Select “File: Connect”





Appendix: Linksys Software Controls

Select "View Temperature Profile" to setup multiple-step temperature ramps



A previously-set temperature profile may affect your ability to control the stage. If control difficulties occur, check the temperature profile settings.

Profile

Profile - Cycle mode off

Ramp	Rate	Limit	Time	Delay
1	1	20.0	30	-
2	0	0.0	0	-
3	0	0.0	0	-
4	0	0.0	0	-
5	0	0.0	0	-
6	0	0.0	0	-
7	0	0.0	0	-
8	0	0.0	0	-
9	0	0.0	0	-
10	0	0.0	0	-

If you are using the DSC, you may be required to use a Temperature Profile!!!